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Indian Standard

AMMONIUM NITRATE FOR NITROUS OXIDE PRODUCTION — SPECIFICATION

UDC 661·525 : 546·172·5

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

July 1992 **Price Group 4**

General Inorganic Chemicals Sectional Committee, CHD 003

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

Ammonium nitrate is extensively used both in commercial and military explosives. Under controlled condition, ammonium nitrite is used for the manufacture of nitrous oxide which is an important anaesthetic gas. Ammonium nitrate prills/crystals produced from non-conventional sources such as ammonium nitrate liquor from phosphatic fertilizer industry or any such other sources should not be employed for the production of any anaesthetic grade nitrous oxide gas.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this standard.

Indian Standard

AMMONIUM NITRATE FOR NITROUS OXIDE PRODUCTION — SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for ammonium nitrate for nitrous oxide production.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

3.1 Description

The material shall be in the form of prills or crystals, as agreed to between the purchaser and the supplier. It shall be colourless or milk white and free from grit, foreign matter and visible impurities.

3.2 The material shall be free from anti-caking agents of organic and inorganic nature. There shall be no frothing or darkening on the molten material when the material is heated. The aqueous solution shall be clear, free from sediments, suspended solids and floating particles.

3.3 The material shall comply with the requirements specified in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in column 4 of the table.

3.4 The molten material shall be clear and colourless and no colour shall appear on cooling the molten material when tested according to the method prescribed in Annex B.

4 PACKING AND MARKING

4.1 The material shall be packed in polyethylene lined bags or in such other containers as agreed to between the purchaser and the supplier. The thickness of the polyethylene sheet shall not be less than 0·05 mm.

4.2 Marking

Each package shall be legibly and indelibly marked with the following information:

a) Name and grade of the material,
b) Mass of the material in the package,
c) Indication of the source of manufacture,
d) Month and year of manufacture, and
e) Lot number/batch number.

5 SAMPLING

The procedure for drawing representative samples from a lot and the criteria for conformity of the material to the requirements of this specification shall be as prescribed in Annex C.

Table 1 Requirements for Ammonium Nitrate for Nitrous Oxide Production

(*Clause* 3.3)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Clause No. in Annex A)
(1)	(2)	(3)	(4)
i)	Purity, percent by mass, *Min*	99·0	A-2
ii)	Moisture, percent by mass, *Max*	1·0	A-3
iii)	Chlorides (as Cl), percent by mass, *Max*	0·001	A-4
iv)	Sulphates (as SO_4), percent by mass, *Max*	0·024	A-5
v)	Nitrites (as ammonium nitrite), percent by mass, *Max*	NIL	A-6
vi)	Non-volatile matter, percent by mass, *Max*	0·05	A-7
vii)	Iron (as Fe), percent by mass, *Max*	0·000 5	A-8
viii)	Organic matter, percent by mass, *Max*	NIL	A-9
ix)	*p*H of 5 percent aqueous solution	5·0 - 5·5	A-10

ANNEX A

(*Clause* 3.3)

ANALYSIS OF AMMONIUM NITRATE FOR NITROUS OXIDE PRODUCTION

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 DETERMINATION OF PURITY

A-2.0 Outline of the Method

Standard potassium hydroxide solution is added to ammonium nitrate solution and the excess of alkali is back titrated with standard sulphuric acid.

$$NH_4NO_3 + KOH \rightarrow KNO_3 + NH_3 + H_2O$$

A-2.1 Reagents

A-2.1.1 *Potassium Hydroxide* — 0·5 N.

A-2.1.2 *Sulphuric Acid* — 0·5 N.

A-2.1.3 *Methyl Red Indicator* — 0·1 percent in ethyl alcohol.

A-2.2 Procedure

Weigh 1·0 g of the material and transfer to a 500-ml conical flask. Dissolve the material in about 100 ml of water. Add 50 ml of 0·5 N potassium hydroxide solution and boil almost to dryness on the hot plate to remove ammonia. Place a glass funnel in the neck of the conical flask to prevent splashing. Dilute the residual solution and titrate with standard 0·5 N sulphuric acid, using methyl red as indicator. Carry out a blank titration using 50 ml of 0·5 N potassium hydroxide solution. Subtract the volume of standard sulphuric acid consumed in blank titration from that required in sample titration to calculate the volume in ml of standard sulphuric acid required for the titration (V).

A-2.3 Calculation

$$\text{Ammonium nitrate, percent by mass} = \frac{V \times N \; 8{\cdot}002}{M}$$

where

V = volume in ml of standard sulphuric acid required for the titration,

N = normality of sulphuric acid, and

M = mass in g of the material taken for the test.

A-3 DETERMINATION OF MOISTURE

A-3.1 Apparatus

A-3.1.1 *Air Oven*

A-3.1.2 *Petri Dish* — of diameter 80 mm and height 15 mm.

A-3.2 Procedure

Weigh accurately 5 g of the material in a petri dish and spread it uniformly by gentle tapping. Care should be taken to ensure that prills form a monolayer. Place the dish containing the test sample in an air oven maintained at 100 ± 2°C for 25-35 minutes (*see* Note 1). Remove the dish from the oven and cool it in a sulphuric acid/calcium chloride desiccator and weigh to constant mass.

NOTE — This method is not suitable for impure ammonium nitrate containing sensitizing impurities such as chlorides, chromates, organic matters, etc, as these may initiate decomposition of the material at the drying temperature. In such cases the drying should be carried out in a vacuum oven at 75°C for two hours.

A-3.3 Calculation

$$\text{Moisture, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of the material and dish before heating,

M_2 = mass in g of the material and dish after heating, and

M = mass in g of the material taken for the test.

A-4 DETERMINATION OF CHLORIDES

A-4.0 Outline of the Method

Chlorides are determined by comparing the opalescence produced by silver nitrate solution against that of a standard chloride solution in Nessler cylinders.

A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders* — 50-ml capacity.

A-4.2 Reagents

A-4.2.1 *Dilute Nitric Acid* — approximately 5 N.

A-4.2.2 *Silver Nitrate Solution* — approximately 5 N.

A-4.2.3 *Standard Chloride Solution*

Dissolve 0·165 g of sodium chloride, dried at 105 ± 2°C, in water and make up to 1 000 ml. One millilitre of this solution contains 0·1 mg of chloride (as Cl).

A-4.3 Procedure

Weigh accurately 17·5 g of the material in a 50-ml Nessler cylinder and dissolve in a minimum quantity of chloride free water. Add 10 ml of dilute nitric acid followed by 1 ml of silver nitrate solution and dilute to the mark. Stir with a glass rod and set aside for 5 minutes. In another Nessler cylinder add 1·75 ml of standard chloride solution followed by other reagents except the sample solution and treat in the similar manner.

A-4.3.1 Any turbidity produced in the sample solution shall not be greater than that produced in the standard chloride solution.

A-5 DETERMINATION OF SULPHATES

A-5.0 Two turbidimetric methods are prescribed. The first method is for routine analysis. The second method shall be used in case of dispute.

A-5.1 First Method

A-5.1.1 *Reagents*

A-5.1.1.1 *Hydrochloric acid* — 10 percent (*m/v*).

A-5.1.1.2 *Barium chloride solution* — 10 percent (*m/v*).

A-5.1.1.3 *Sulphuric acid* — 0·01 N.

A-5.1.2 *Procedure*

Weigh accurately 5 g of material and dissolve in water. Transfer the solution to a 50-ml Nessler cylinder. Add 10 ml of dilute hydrochloric acid and 1 ml of barium chloride solution and dilute to the mark. Stir immediately with a glass rod and set aside for 5 minutes. In another Nessler cylinder add 2·5 ml of 0·01 N sulphuric acid followed by other reagents except the sample and treat in the same manner.

A-5.1.2.1 Any turbidity produced in the sample solution shall not be greater than that produced in the standard sulphate solution.

A-5.2 Second Method

A-5.2.1 *Outline of the Method*

Sulphates are determined by comparing the turbidity produced by barium sulphate solution, against a standard turbidity in Nessler cylinders.

A-5.2.2 *Apparatus*

A-5.2.2.1 *Nessler cylinders* — 50-ml capacity.

A-5.2.3 *Reagents*

A-5.2.3.1 *Sodium carbonate* — solid.

A-5.2.3.2 *Dilute hydrochloric acid* — approximately 2 N.

A-5.2.3.3 *Standard sulphuric acid* — 0·002 N.

A-5.2.3.4 *Barium sulphate reagent*

Dissolve 2 g of barium chloride in 75 ml of water, add 20 ml of 95 percent (*v/v*) ethyl alcohol and 5 ml of standard sulphuric acid. It shall be prepared afresh.

A-5.2.4 *Procedure*

A-5.2.4.1 Weigh accurately 10 g of the prepared sample into a porcelain dish and dissolve in 40 ml of water. Add 0·05 g of sodium carbonate and heat on a sand bath to about 120°C to volatilize water and heat over a very low flame until the evolution of white fumes has ceased. Finally heat the dish at 600°C in a furnace for 15 minutes. Cool, dissolve the residue in 10 ml of water, add 1 ml of dilute hydrochloric acid and filter through a Whatman No. 41 filter paper. Transfer the filtrate to a Nessler cylinder, add 10 ml of water and 1 ml of barium sulphate reagent. Mix well and allow to stand for 5 minutes.

A-5.2.4.2 Prepare a series of sulphate standard turbidities with different known volumes of standard sulphuric acid and 1 ml of barium sulphate reagent in a similar manner and compare the turbidities. Calculate the sulphate content (as SO_4) in the sulphate standard in which the turbidity most closely matches with that in the solution of the material.

A-6 DETERMINATION OF NITRITE

A-6.0 Two methods are prescribed, namely colorimetric method and volumetric method. Colorimetric method is recommended for routine analysis. In case of dispute, volumetric method shall be the referee method.

A-6.1 Colorimetric Method

A-6.1.1 *Outline of the Method*

When dilute solutions of sulphanilic acid and α-naphthylamine are treated with nitrous acid, a red colouration is produced which may be used for the colorimetric determination of nitrites. The full colour does not appear at once. A satisfactory colour comparison can be made after 10-15 minutes provided the sample and standard are treated in the same manner.

A-6.1.2 *Reagents*

A-6.1.2.1 *Sulphanilic acid reagent (solution 1)*

Dissolve 0·6 g of sulphanilic acid in 100 ml of 20 percent (*v/v*) dilute hydrochloric acid.

A-6.1.2.2 *α-Naphthylamine reagent (solution 2)*

Dissolve 0·48 g of α-naphthylamine in 100 ml of 20 percent (*v/v*) dilute hydrochloric acid.

A-6.1.2.3 *Standard nitrite solution*

Dissolve 4·93 g of sodium nitrite in water and dilute with deionized water to 1 000 ml. Dilute 10 ml of standard solution to 1 000 ml with deionized water.

A-6.1.2.4 *Sodium acetate solution* — 2 M

Dissolve 16·4 g of anhydrous sodium acetate in water and dilute to 100 ml with water.

A-6.1.3 *Procedure*

Place the quantity of the sample containing about 0·03 mg or less of nitrite in a 50-ml volumetric flask. Add 1 ml of sulphanilic acid reagent, mix well and allow to stand at least for 3 minutes but not more than 10 minutes at room temperature in diffused light. Introduce 1 ml of α-naphthylamine reagent and 1 ml of 2 M sodium acetate solution to act as buffer (*p*H 2 to 2·5). Dilute to the mark and mix well. Use a photo-electric colorimeter with a green filter and measure transmittance. Construct a reference curve with known nitrite standards, preferably in the nitrite nitrogen range of the sample and measure the concentration of the nitrite in the sample solution from the reference curve.

A-6.1.4 *Calculation*

The concentration of nitrite is directly read from the reference curve and calculated as ammonium nitrite.

A-6.2 Volumetric Method

A-6.2.1 *Reagents*

A-6.2.1.1 *Potassium permanganate* — 0·02 N.

A-6.2.1.2 *Sulphuric acid* — 1 : 1 (*v/v*).

A-6.2.2 *Procedure*

A-6.2.2.1 Weigh accurately about 30 g of the sample and transfer to a 100-ml volumetric flask and dissolve by shaking in 40 ml of water. Make up the volume to the mark with water. Stopper the flask and shake well. Take up the solution in a burette.

A-6.2.2.2 Take 2·5 ml of freshly prepared 0·02 N potassium permanganate solution in a conical flask. Add 5 ml of sulphuric acid (1 : 1). Dilute to 40 ml with water, heat to around 60°C and titrate against sample solution taken in the burette till the pink colour disappears.

A-6.2.3 *Calculation*

$$\text{1 ml of 1 N potassium permanganate} = \text{0·032 0 g of ammonium nitrite}$$

A-7 DETERMINATION OF NON-VOLATILE MATTER

A-7.1 Procedure

Weigh accurately about 5 g of the material in a small porcelain dish and heat over a low flame until the material has completely decomposed. Heat the residue to a dull redness at about 800°C for one hour to constant mass. Cool the dish in a desiccator and weigh. Report the residue as non-volatile matter of the original sample.

A-7.2 Calculation

$$\text{Non-volatile matter, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = mass in g of the residue; and

M = mass in g of the material taken for the test.

A-8 DETERMINATION OF IRON

A-8.1 Outline of the Method

Ferric (but not ferrous) iron reacts with thiocyanate to give a series of intensely red coloured complexes which remain in true solution. In the colorimetric determination, excess of thiocyanate should be used since this increases the intensity and also stability of the colour. Strong acid (hydrochloric or nitric acid concentration 0·052 to 0·05 M) should be present to suppress the hydrolysis of ferric iron.

A-8.2 Reagents

A-8.2.1 *Potassium Thiocyanate Solution*

Dissolve 20 g of potassium thiocyanate in 100 ml of water.

A-8.2.2 *Concentrated Nitric Acid*

(*See* IS 264 : 1976).

A-8.2.3 *Standard Iron (Ferric Solution)*

Dissolve 0·864 g of ferric ammonium sulphate in water, add 10 ml of concentrated nitric acid and dilute it to 1 000 ml. One ml of this solution is equivalent to 0·1 mg of iron (as Fe).

A-8.2.4 *Potassium Permanganate Solution*

0·2 percent (*m/v*).

A-8.3 Procedure

Dissolve accurately 5 g of the material in 1 : 1 water-nitric acid mixture and evaporate nearly to dryness to expel excess of acid. Dilute slightly with water, oxidize any ferrous iron to the ferric state with dilute potassium permanganate solution (0·2 percent), adding slowly dropwise until a slight pink colour remains after stirring well.

Make up the solution to 250 ml. Place 50 ml of the solution in a Nessler cylinder, add 5 ml of thiocyanate solution and 2 to 4 ml of 4 N nitric acid. Add the same amounts of reagents to 50 ml of water contained in a similar Nessler cylinder and run in standard iron solution from burette (use long glass rod with flattened end for mixing) until colours are matched. Note the exact volume (X ml) of the standard iron solution added. Repeat determination using (50 — X) ml of water. Comparison of the standard and unknown should be made soon after preparation since the colour may fade on standing for a long time.

NOTE — For estimating the concentration of ferric iron more accurately, the use of photoelectric colorimeter may be resorted to. A filter showing maximum transmission near 480 mm should be used. Standard solutions of ferric iron which are in the range of the concentration of ferric iron likely to be present in the sample are prepared and the transmittance of each such solution measured with photoelectric colorimeter. A reference curve is then prepared by plotting the concentration versus transmittance. Now the transmittance of the sample solution is also measured and from the reference curve, the concentration of ferric iron as $Fe(NO_3)_3$ is directly read.

A-8.4 Calculation

$$\text{Ferric iron [as } Fe(NO_3)_3 \text{], percent by mass} = \frac{m \times 100}{M}$$

where

m = mass in g of ferric iron as $Fe(NO_3)_3$ as found out from the reference curve, and

M = mass in g of the material taken for the test.

A-9 DETERMINATION OF ORGANIC MATTER

A-9.1 Procedure

Weigh about 50 g of the sample accurately in a tall sintered-glass grade 2 thimble with a small hole at the top of wall for tying a small piece of wire. Put the thimble gently in the soxholet extraction chamber with the help of the wire. Take 200 ml of the suitable solvent (dichloromethane, diethyl ether, acetone, *N*-butyl acetate, etc) in a flack containing a few glass beads. Fit the apparatus on a boiling water bath and continue the extraction for three hours. Remove the condenser and take out the thimble. Replace the condenser and continue boiling till majority of the solvent is recovered in the flask to a tared flat bottomed glass basin with cower. Rinse the flask twice with the suitable solvent and transfer in the basin. Put the basin in a boiling water bath and evaporate the solvent. Place the basin in a water bath maintained at 95°±2°C for two hours. At the end of this period, replace the cover, cool the basin and the cover in a desiccator to room temperature and weigh. Repeat the drying operation till constant mass is obtained.

NOTE — When required the manufacturers shall inform about the name of the solvent to be used.

A-9.2 Calculation

$$\text{Organic matter, percent by mass} = \frac{M_2}{M_1} \times 100$$

where

M_2 = mass in g of the residue after complete evaporation of the solvent extract, and

M_1 = mass in g of the material taken for the test.

A-10 DETERMINATION OF *p*H

A-10.1 Procedure

Dissolve 5 g of the material in 100 ml of water and measure the *p*H of the solution by means of suitable *p*H meter using glass electrode.

ANNEX B

(*Clause* 3.4)

MELT TEST OF AMMONIUM NITRATE FOR NITROUS OXIDE PRODUCTION

B-1 APPARATUS

B-1.1 Porcelain Dish

B-1.2 Bunsen Burner

B-2 PROCEDURE

Take 25 g of the material in a clean, dry, white porcelain dish. Heat it on a Bunsen burner slowly to melting. Observe the melt for clarity and colour. The molten material shall be clear and colourless. On cooling the molten mass, no colour shall appear.

ANNEX C

(*Clause* 5)

SAMPLING OF AMMONIUM NITRATE FOR NITROUS OXIDE PRODUCTION

C-1 GENERAL REQUIREMENTS OF SAMPLING

C-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

C-1.1 Samples shall not be drawn in an exposed place.

C-1.2 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

C-1.3 To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

C-1.4 The samples shall be placed in clean, dry, air-tight glass or other suitable containers which have no action on the material.

C-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

C-1.6 Each sample container shall be sealed air-tight with a stopper after filling, and marked with all particulars of the material (*see* **4.2**) and the date of sampling.

C-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the ambient temperature.

C-2 SCALE OF SAMPLING

C-2.1 Lot

In a single consignment of the material all the containers of the same size and drawn from the same batch of manufacture shall constitute a lot If a consignment is known to consist of different batches of manufacture or of different types and sizes of containers, the containers belonging to the same batch and size shall be grouped together and each such group shall constitute a separate lot.

C-2.1.1 For ascertaining the conformity of the material in a lot to the requirements of the specification, tests shall be carried out for each lot separately. The number of containers to be selected at random from lots of different sizes shall be in accordance with Table 2.

Table 2 Scales of Sampling

(*Clause* C-2.1.1)

Lot Size (N) (1)	Sample Size (n) (2)
Up to 15	3
16 to 25	4
26 to 50	5
51 to 100	7
101 and above	10

C-2.1.2 The containers shall be selected at random from the lot and in order to ensure randomness of selection random number tables shall be used (*see* IS 4905 : 1968). In case such tables are not available, the following procedure is recommended for use:

> "Arrange the containers in the lot in a systematic order and starting from any one container, count them as 1, 2, 3,.......... etc, up to r and so on, where r is the integral part of N/n (N and n being the lot size and sample size respectively). Every rth container thus counted shall be withdrawn to constitute the sample."

C-3 PREPARATION OF TEST SAMPLES

C-3.1 From each of the containers selected, draw a small representative portion of the material not less than 100 g in mass.

C-3.2 Out of these portions, small but equal quantity of the material shall be taken and mixed thoroughly, to form a composite sample of mass not less than 150 g. The composite sample shall be divided into 3 equal parts, one for the purchaser, another for the supplier and the third to be used as a referce sample.

C-3.3 The remaining portion of the material from each container shall be divided into three equal parts and transferred to separate bottles, giving full identification particulars of the samples on the bottle. The material in each bottle constitutes an individual sample. One of these three sets (each set containing one bottle representing each container sampled) shall be marked for the purchaser, another for the supplier and the third for the referee.

C-3.4 The referee test sample consisting of a composite sample and a set of individual samples shall bear the seal of both the purchaser and the supplier. They shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

C-4 NUMBER OF TEST

C-4.1 Test for determination of moisture shall be performed on each of the individual samples.

C-4.2 Tests for the determination of all the remaining characteristics given in **3** and Table 1 shall be carried out on the composite sample.

C-5 CRITERIA FOR CONFORMITY

C-5.1 From each set of the individual test results for moisture, the average ($\bar{X}$) and the range (R) of the test results shall be computed (range R is defined as the difference between the maximum and the minimum values of the test results).

C-5.2 For declaring the conformity of the lot to the specification, requirements of all other characteristics tested on the composite sample, the test results shall meet the corresponding requirements specified in **3** and Table 1.

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